Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-Bromo-1,3-dimethyl-1*H*-imidazo-[4,5-*b*]pyridin-2(3*H*)-one

Siham Dahmani,[a] Youssef Kandri Rodi,[a] Hafid Zouihri,[b] El Mokhtar Essassi[c] and Seik Weng Ng[d]*

[a]Laboratoire de Chimie Organique Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fès, Morocco, [b]CNRST Division UATRS, Angle Allal Fassi/FAR, BP 8027 Hay Riad, Rabat, Morocco, [c]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

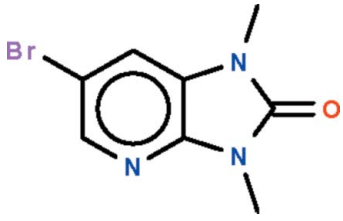
Received 24 June 2010; accepted 26 June 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.086; data-to-parameter ratio = 21.9.

The non-H atoms of the two independent molecules in the asymmetric unit of the title compound, $C_8H_8BrN_3O$, are planar (r.m.s. deviations = 0.015 and 0.019 Å). In the crystal, the molecules are linked into a zigzag chain along the c axis by C—H···O hydrogen bonds.

Related literature

For the synthesis of the un-brominated compound, see: Yutilov *et al.* (1998, 2005).



Experimental

Crystal data

$C_8H_8BrN_3O$ M_r = 242.08
Monoclinic, $P2_1/c$
a = 21.7981 (4) Å
b = 3.9929 (1) Å
c = 20.6636 (3) Å
β = 95.398 (1)°
V = 1790.53 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 4.55 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker X8 APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.396, T_{max} = 0.548
21586 measured reflections
5229 independent reflections
3539 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.01
5229 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8*C*···O2^{i}	0.96	2.33	3.279 (3)	167
C11—H11···O1	0.93	2.49	3.321 (3)	148

Symmetry code: (i) $x, -y+\frac{5}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5119).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA..
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**. Submitted.
Yutilov, Yu. M., Lopatinskaya, Kh. Ya., Smolyar, N. N. & Gres'ko, S. V. (2005). *Russ. J. Org. Chem.* **41**, 575–579.
Yutilov, Yu. M., Malyutina, V. F., Lopatinskaya, Kh. Ya. & Svertilova, I. A. (1998). *Russ. J. Org. Chem.* **34**, 1363–1364.

supporting information

Acta Cryst. (2010). E**66**, o1904 [https://doi.org/10.1107/S1600536810025134]

6-Bromo-1,3-dimethyl-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

Siham Dahmani, Youssef Kandri Rodi, Hafid Zouihri, El Mokhtar Essassi and Seik Weng Ng

S1. Comment

The imidazo[4,5-*b*]pyridine unit is an important heterocyclic nucleus found in a large number of medicinal compounds. The synthesis of 1,3-dimethyl-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one involves several steps (Yutilov *et al.*, 1998, 2005). The 6-bromo derivative (Scheme I) is conveniently synthesized by the direct reaction of 6-bromo-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one with methyl iodide.

The aysmmetric unit (Fig. 1) has two independent molecules. Both are planar.

S2. Experimental

To a mixture of 6-bromo-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one (0.15 g, 1 mmol), potassium carbonate (0.38 g, 4 mmol) and tetra-*n*-butylammonium bromide (0.02 g, 0.1 mmol) in DMF (10 ml) was added methyl iodide (0.1 ml, 2.5 mmol). Stirring was continued at room temperature for 12 h. After the completion of reaction (as monitored by TLC), the mixture was filtered and the solvent removed under reduced pressure. The residue was purified by column chromatography on silica gel with ethyl acetate-hexane (1:3) as eluent. The compound was recrystallized from ethyl acetate-hexane (1:3) to afford colourless crystals.

S3. Refinement

H atoms were placed in calculated positions (C-H = 0.93–0.96 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C).

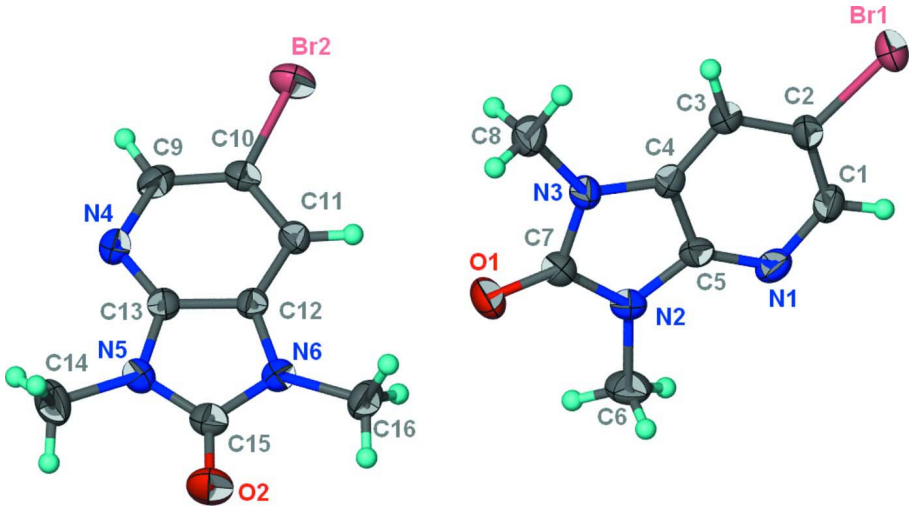


Figure 1

Displacement ellipsoid plot (Barbour, 2001) of the two independent molecules of $C_8H_8BrN_3O$ at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

6-Bromo-1,3-dimethyl-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

Crystal data

$C_8H_8BrN_3O$
$M_r = 242.08$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 21.7981$ (4) Å
$b = 3.9929$ (1) Å
$c = 20.6636$ (3) Å
$\beta = 95.398$ (1)°
$V = 1790.53$ (6) Å^3
$Z = 8$

$F(000) = 960$
$D_x = 1.796$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6375 reflections
$\theta = 2.6$–25.2°
$\mu = 4.55$ mm^{-1}
$T = 293$ K
Prism, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker X8 APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.396$, $T_{max} = 0.548$

21586 measured reflections
5229 independent reflections
3539 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.6°$
$h = -30\rightarrow28$
$k = -5\rightarrow5$
$l = -29\rightarrow28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.086$
$S = 1.01$
5229 reflections
239 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.1144P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.407800 (11)	0.91753 (7)	0.468246 (11)	0.05023 (9)
N1	0.44555 (8)	0.5071 (5)	0.65032 (9)	0.0416 (4)
N2	0.37942 (8)	0.4903 (5)	0.73723 (8)	0.0400 (4)
N3	0.29799 (8)	0.7480 (5)	0.68786 (8)	0.0387 (4)
N4	0.05385 (8)	0.5642 (5)	0.92033 (9)	0.0400 (4)
N5	0.12070 (8)	0.8692 (5)	0.99889 (8)	0.0391 (4)
N6	0.19961 (8)	0.9449 (5)	0.94033 (8)	0.0373 (4)
O1	0.29345 (8)	0.5668 (5)	0.79455 (8)	0.0568 (5)
O2	0.20597 (8)	1.1650 (5)	1.04514 (8)	0.0576 (5)
C1	0.44708 (10)	0.6147 (6)	0.58839 (11)	0.0421 (5)
H1	0.4826	0.5767	0.5678	0.050*
C2	0.39881 (10)	0.7769 (6)	0.55465 (10)	0.0374 (5)
C3	0.34349 (10)	0.8436 (5)	0.58128 (10)	0.0356 (5)
H3	0.3105	0.9528	0.5585	0.043*
C4	0.34205 (9)	0.7335 (5)	0.64419 (10)	0.0339 (4)
C5	0.39361 (9)	0.5704 (5)	0.67562 (10)	0.0345 (5)
C6	0.42023 (12)	0.3314 (7)	0.78847 (13)	0.0546 (7)
H6A	0.3960	0.2231	0.8188	0.082*
H6B	0.4460	0.4983	0.8107	0.082*
H6C	0.4455	0.1684	0.7696	0.082*
C7	0.32072 (10)	0.5976 (6)	0.74587 (11)	0.0411 (5)
C8	0.23694 (10)	0.8923 (7)	0.67783 (13)	0.0503 (6)
H8A	0.2289	1.0214	0.7153	0.075*
H8B	0.2070	0.7164	0.6713	0.075*
H8C	0.2344	1.0348	0.6402	0.075*
C9	0.05044 (10)	0.4500 (6)	0.85870 (11)	0.0415 (5)
H9	0.0149	0.3385	0.8423	0.050*
C10	0.09723 (10)	0.4905 (5)	0.81873 (10)	0.0356 (5)
C11	0.15197 (9)	0.6529 (5)	0.83943 (10)	0.0350 (5)
H11	0.1839	0.6795	0.8130	0.042*
C12	0.15516 (9)	0.7711 (5)	0.90210 (9)	0.0311 (4)
C13	0.10513 (9)	0.7226 (5)	0.93943 (10)	0.0337 (4)
C14	0.08264 (13)	0.8834 (8)	1.05313 (13)	0.0621 (8)
H14A	0.1083	0.8604	1.0933	0.093*
H14B	0.0530	0.7048	1.0492	0.093*
H14C	0.0615	1.0945	1.0526	0.093*
C15	0.17830 (10)	1.0112 (6)	0.99985 (11)	0.0408 (5)
C16	0.25863 (10)	1.0616 (6)	0.92138 (12)	0.0474 (6)
H16A	0.2842	1.1318	0.9593	0.071*
H16B	0.2520	1.2468	0.8919	0.071*
H16C	0.2786	0.8829	0.9004	0.071*

Br2	0.086179 (12)	0.32023 (7)	0.732957 (11)	0.05183 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05040 (16)	0.06094 (17)	0.04140 (14)	−0.00344 (12)	0.01514 (11)	0.00544 (11)
N1	0.0294 (9)	0.0492 (12)	0.0453 (11)	0.0036 (8)	−0.0008 (8)	−0.0047 (8)
N2	0.0366 (10)	0.0488 (12)	0.0335 (10)	0.0022 (8)	−0.0025 (8)	0.0032 (8)
N3	0.0332 (10)	0.0493 (11)	0.0343 (9)	0.0062 (8)	0.0063 (7)	0.0052 (8)
N4	0.0318 (10)	0.0484 (11)	0.0403 (10)	−0.0036 (8)	0.0061 (8)	0.0035 (8)
N5	0.0396 (10)	0.0478 (11)	0.0306 (9)	0.0006 (8)	0.0071 (8)	−0.0029 (8)
N6	0.0332 (9)	0.0425 (10)	0.0357 (9)	−0.0038 (8)	0.0012 (7)	0.0002 (8)
O1	0.0547 (10)	0.0782 (13)	0.0395 (9)	0.0077 (9)	0.0152 (8)	0.0125 (8)
O2	0.0627 (12)	0.0644 (12)	0.0439 (10)	−0.0092 (9)	−0.0042 (9)	−0.0157 (8)
C1	0.0303 (11)	0.0507 (14)	0.0463 (13)	−0.0013 (10)	0.0096 (10)	−0.0058 (10)
C2	0.0362 (12)	0.0429 (12)	0.0337 (11)	−0.0065 (10)	0.0069 (9)	−0.0025 (9)
C3	0.0322 (11)	0.0394 (12)	0.0350 (11)	0.0000 (9)	0.0015 (9)	0.0010 (8)
C4	0.0289 (11)	0.0356 (11)	0.0373 (11)	−0.0003 (9)	0.0045 (8)	−0.0016 (9)
C5	0.0332 (11)	0.0370 (11)	0.0322 (11)	−0.0027 (9)	−0.0028 (8)	−0.0038 (8)
C6	0.0565 (16)	0.0608 (17)	0.0441 (13)	0.0095 (13)	−0.0084 (11)	0.0098 (12)
C7	0.0400 (12)	0.0477 (13)	0.0357 (12)	0.0000 (10)	0.0041 (9)	0.0006 (9)
C8	0.0385 (13)	0.0615 (16)	0.0520 (14)	0.0104 (11)	0.0101 (11)	0.0106 (12)
C9	0.0333 (12)	0.0433 (13)	0.0463 (13)	−0.0043 (10)	−0.0046 (10)	0.0021 (10)
C10	0.0405 (12)	0.0358 (12)	0.0295 (10)	0.0029 (9)	−0.0013 (9)	0.0024 (8)
C11	0.0338 (11)	0.0393 (12)	0.0321 (11)	0.0031 (9)	0.0048 (8)	0.0051 (8)
C12	0.0293 (10)	0.0320 (11)	0.0318 (10)	0.0012 (8)	0.0013 (8)	0.0039 (8)
C13	0.0321 (11)	0.0362 (11)	0.0327 (10)	0.0040 (9)	0.0028 (8)	0.0046 (8)
C14	0.0624 (17)	0.082 (2)	0.0457 (14)	−0.0051 (15)	0.0220 (13)	−0.0141 (13)
C15	0.0435 (13)	0.0423 (13)	0.0357 (11)	0.0036 (10)	−0.0010 (10)	0.0011 (9)
C16	0.0336 (12)	0.0531 (14)	0.0557 (15)	−0.0068 (11)	0.0049 (10)	0.0010 (11)
Br2	0.06391 (18)	0.05326 (16)	0.03660 (13)	−0.00056 (12)	−0.00434 (11)	−0.00705 (10)

Geometric parameters (Å, º)

Br1—C2	1.899 (2)	C3—C4	1.375 (3)
N1—C5	1.316 (3)	C3—H3	0.93
N1—C1	1.353 (3)	C4—C5	1.404 (3)
N2—C5	1.376 (3)	C6—H6A	0.96
N2—C7	1.377 (3)	C6—H6B	0.96
N2—C6	1.462 (3)	C6—H6C	0.96
N3—C4	1.380 (3)	C8—H8A	0.96
N3—C7	1.390 (3)	C8—H8B	0.96
N3—C8	1.447 (3)	C8—H8C	0.96
N4—C13	1.313 (3)	C9—C10	1.381 (3)
N4—C9	1.348 (3)	C9—H9	0.93
N5—C13	1.374 (3)	C10—C11	1.390 (3)
N5—C15	1.376 (3)	C10—Br2	1.893 (2)
N5—C14	1.457 (3)	C11—C12	1.374 (3)

N6—C12	1.379 (3)	C11—H11	0.93
N6—C15	1.381 (3)	C12—C13	1.407 (3)
N6—C16	1.456 (3)	C14—H14A	0.96
O1—C7	1.222 (3)	C14—H14B	0.96
O2—C15	1.229 (3)	C14—H14C	0.96
C1—C2	1.369 (3)	C16—H16A	0.96
C1—H1	0.93	C16—H16B	0.96
C2—C3	1.397 (3)	C16—H16C	0.96

C5—N1—C1	114.43 (18)	N2—C7—N3	106.43 (18)
C5—N2—C7	109.90 (17)	N3—C8—H8A	109.5
C5—N2—C6	126.63 (19)	N3—C8—H8B	109.5
C7—N2—C6	123.40 (19)	H8A—C8—H8B	109.5
C4—N3—C7	109.41 (18)	N3—C8—H8C	109.5
C4—N3—C8	127.62 (19)	H8A—C8—H8C	109.5
C7—N3—C8	122.96 (19)	H8B—C8—H8C	109.5
C13—N4—C9	114.63 (18)	N4—C9—C10	123.2 (2)
C13—N5—C15	109.67 (18)	N4—C9—H9	118.4
C13—N5—C14	126.4 (2)	C10—C9—H9	118.4
C15—N5—C14	123.9 (2)	C9—C10—C11	122.1 (2)
C12—N6—C15	109.26 (17)	C9—C10—Br2	118.81 (16)
C12—N6—C16	126.73 (18)	C11—C10—Br2	119.06 (16)
C15—N6—C16	123.92 (19)	C12—C11—C10	114.59 (19)
N1—C1—C2	123.1 (2)	C12—C11—H11	122.7
N1—C1—H1	118.5	C10—C11—H11	122.7
C2—C1—H1	118.5	N6—C12—C11	133.1 (2)
C1—C2—C3	122.7 (2)	N6—C12—C13	107.11 (17)
C1—C2—Br1	118.44 (16)	C11—C12—C13	119.8 (2)
C3—C2—Br1	118.88 (16)	N4—C13—N5	127.28 (19)
C4—C3—C2	114.1 (2)	N4—C13—C12	125.70 (19)
C4—C3—H3	122.9	N5—C13—C12	107.02 (18)
C2—C3—H3	122.9	N5—C14—H14A	109.5
C3—C4—N3	133.2 (2)	N5—C14—H14B	109.5
C3—C4—C5	119.79 (19)	H14A—C14—H14B	109.5
N3—C4—C5	107.04 (18)	N5—C14—H14C	109.5
N1—C5—N2	126.9 (2)	H14A—C14—H14C	109.5
N1—C5—C4	125.9 (2)	H14B—C14—H14C	109.5
N2—C5—C4	107.22 (18)	O2—C15—N6	126.3 (2)
N2—C6—H6A	109.5	O2—C15—N5	126.8 (2)
N2—C6—H6B	109.5	N6—C15—N5	106.92 (18)
H6A—C6—H6B	109.5	N6—C16—H16A	109.5
N2—C6—H6C	109.5	N6—C16—H16B	109.5
H6A—C6—H6C	109.5	H16A—C16—H16B	109.5
H6B—C6—H6C	109.5	N6—C16—H16C	109.5
O1—C7—N2	127.3 (2)	H16A—C16—H16C	109.5
O1—C7—N3	126.3 (2)	H16B—C16—H16C	109.5

C5—N1—C1—C2	0.2 (3)	C13—N4—C9—C10	0.9 (3)

N1—C1—C2—C3	−0.4 (4)	N4—C9—C10—C11	0.2 (3)
N1—C1—C2—Br1	179.39 (17)	N4—C9—C10—Br2	−179.59 (17)
C1—C2—C3—C4	0.3 (3)	C9—C10—C11—C12	−0.6 (3)
Br1—C2—C3—C4	−179.52 (15)	Br2—C10—C11—C12	179.19 (15)
C2—C3—C4—N3	179.6 (2)	C15—N6—C12—C11	178.1 (2)
C2—C3—C4—C5	0.0 (3)	C16—N6—C12—C11	1.5 (4)
C7—N3—C4—C3	−179.7 (2)	C15—N6—C12—C13	−1.1 (2)
C8—N3—C4—C3	0.5 (4)	C16—N6—C12—C13	−177.7 (2)
C7—N3—C4—C5	−0.1 (2)	C10—C11—C12—N6	−179.2 (2)
C8—N3—C4—C5	−179.9 (2)	C10—C11—C12—C13	−0.1 (3)
C1—N1—C5—N2	−179.9 (2)	C9—N4—C13—N5	178.9 (2)
C1—N1—C5—C4	0.1 (3)	C9—N4—C13—C12	−1.7 (3)
C7—N2—C5—N1	180.0 (2)	C15—N5—C13—N4	−179.7 (2)
C6—N2—C5—N1	3.0 (4)	C14—N5—C13—N4	−1.1 (4)
C7—N2—C5—C4	−0.1 (2)	C15—N5—C13—C12	0.8 (2)
C6—N2—C5—C4	−177.1 (2)	C14—N5—C13—C12	179.4 (2)
C3—C4—C5—N1	−0.3 (3)	N6—C12—C13—N4	−179.3 (2)
N3—C4—C5—N1	−180.0 (2)	C11—C12—C13—N4	1.4 (3)
C3—C4—C5—N2	179.80 (19)	N6—C12—C13—N5	0.2 (2)
N3—C4—C5—N2	0.1 (2)	C11—C12—C13—N5	−179.13 (19)
C5—N2—C7—O1	179.8 (2)	C12—N6—C15—O2	−179.2 (2)
C6—N2—C7—O1	−3.1 (4)	C16—N6—C15—O2	−2.4 (4)
C5—N2—C7—N3	0.1 (3)	C12—N6—C15—N5	1.5 (2)
C6—N2—C7—N3	177.2 (2)	C16—N6—C15—N5	178.30 (19)
C4—N3—C7—O1	−179.7 (2)	C13—N5—C15—O2	179.3 (2)
C8—N3—C7—O1	0.1 (4)	C14—N5—C15—O2	0.6 (4)
C4—N3—C7—N2	0.0 (3)	C13—N5—C15—N6	−1.4 (2)
C8—N3—C7—N2	179.8 (2)	C14—N5—C15—N6	179.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*C*···O2^{i}	0.96	2.33	3.279 (3)	167
C11—H11···O1	0.93	2.49	3.321 (3)	148

Symmetry code: (i) *x*, −*y*+5/2, *z*−1/2.